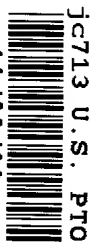


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jc713 U.S. PTO

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PTO/SB/13/PCT (12-97)
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REQUEST FOR FILING A CONTINUATION OR DIVISION OF AN INTERNATIONAL APPLICATION

DOCKET NUMBER	ANTICIPATED CLASSIFICATION OF THIS APPLICATION		PRIOR APPLICATION EXAMINER	ART UNIT
AE97/151US	CLASS	SUBCLASS		

jc584 U.S. PTO
 09/447030
 4477711

Address to:
 Assistant Commissioner for Patents
 Washington, D.C. 20231

This is a request for filing a ☒ continuation ☐ divisional application under 37 CFR 1.53(b), of pending prior international application Number PCTEP/ **98/3161**, filed on **28/5/98** entitled **METHOD FOR PRODUCING SUBSTANTIALLY GLOBULAR LYOGELS AND AEROGELS**

which designated the United States.

Note: 37 CFR 1.53(c) or (d) cannot be used to file a continuation or divisional application of an international application which has not entered the national stage.

CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
TOTAL CLAIMS (37 CFR 1.16(c))	12	- 20 =	0	x \$ _____ =	\$ 0.00
INDEPENDENT CLAIMS (37 CFR 1.16(b))	1	- 3 =	0	x \$ _____ =	0.00
MULTIPLE DEPENDENT CLAIMS (if applicable) (37 CFR 1.16(d))				+ \$ _____ =	0.00
BASIC FEE (37 CFR 1.16(a))					+ 760.00
Total of above Calculations =					0.00
Reduction by 50% for filing by small entity (Note 37 CFR 1.9, 1.27, 1.28).					0.00
TOTAL =					760.00

- Enclosed are the specification, claims and drawing(s).
- ☐ A statement to establish small entity status under 37 CFR 1.9 and 1.27 is enclosed.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required under 37 CFR 1.16 and 1.17, or ☒ credit any overpayment to Deposit Account No. **19-0733**. A duplicate copy of this sheet is enclosed.
- ☐ A check in the amount of \$ _____ is enclosed.
- ☒ Amend the specification by inserting before the first line the sentence: "This application is a ☒ continuation ☐ division of international application number PCT**EP98/03161**, filed **28 May, 1998, now abandoned.** (status, abandoned, pending, etc.)."

[Page 1 of 2]

Burden Hour Statement: This form is estimated to take 0.5 hours to complete. Time will vary depending upon the needs of individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

(REQUEST FOR FILING A CONTINUATION OR DIVISION OF AN INTERNATIONAL APPLICATION, PAGE 2)

6. ☐ A declaration under 37 CFR 1.63 is enclosed.

7. ☒ Priority of foreign application number DE19722738.4 filed on 30MAY1997 (30/05/97) in Germany
is claimed under 35 U.S.C. 119(a) - (d).

☐ The certified copy is enclosed.


8. ☐ A preliminary amendment is enclosed.

9. ☒ Also enclosed: **Return receipt postcard, PTO Form 1449, and a copy of International Patent Appln. No. PCT/EP98/03161 and associated documents**

Address all future correspondence to: (May only be completed by applicant, or attorney or agent of record.)

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Nov. 22, 1999
Date


Signature

Peter D. McDermott

Typed or printed name

I hereby certify that this envelope is being deposited as Express Mail,
Certificate No. EL058486144US, on Nov. 22, 1999 with
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Washington, D.C. 20231.

Date: Nov. 22, 1999

☐ Inventor(s)

☐ Assignee of complete interest. Certification under 37 CFR 3.73(b) is enclosed.

☐ Attorney or agent of record

☒ Filed under 37 CFR 1.34(a)

Registration number if acting under 37 CFR 1.34(a) 29,411


Signature

METHOD FOR PRODUCING SUBSTANTIALLY GLOBULAR LYOGELS AND AEROGELS

The object of the invention is a method of producing substantially globular lyogels and aerogels.

Aerogels, particularly those with a porosity above 60% and a density of less than 0.6 g/cu.cm, display extremely low thermal conductivity and are therefore used as a heat insulating material, as described for example in EP-A-0 171 722. Furthermore, by virtue of their very low refraction index for solid substances, it is known to use them for Cerenkov detectors. Furthermore, by reason of their particular acoustic impedance, the literature describes a possible use as an impedance adaptation means, for example in the alpha sound range. It is also possible for them to be used as carriers for effective substances in pharmacy or agriculture

Aerogels in the broader sense, e.g. in the sense of "gel with air as the dispersion agent" are produced by the drying of a suitable gel. The term "aerogel" in this sense embraces aerogels in the narrower sense, xerogels and cryogels. In this respect, a dried gel is termed an aerogel in the narrower sense when the liquid of the gel is eliminated at above critical temperature and starting from pressures above critical pressure. On the other hand, if the liquid is eliminated from the gel under sub-critical conditions, for example with the formation of a liquid/favour interphase, then the resulting gel is frequently referred to also as a xerogel.

When the term aerogels is used in the present invention, these are aerogels in the broader sense, e.g. in the sense of "gel with air as the dispersion medium "

The term does not include aerogels known from earlier literature and which are obtained for example by precipitation of silicic acid (e.g. DE 3025437, DD 296 898) or which occur as pyrogen silicic acid, e.g. Aerosil™. In these cases, during manufacture, no three-dimensional gel lattice develops which is homogeneous over relatively great distances.

Where aerogels are concerned, it is possible basically to differentiate between inorganic and organic aerogels.

Inorganic aerogels have already been known since 1931 (S. S. Kistler, Nature 1931, 127, 741). Since then, aerogels have been forthcoming from various starting materials. In this respect, for example SiO_2 , TiO_2 , ZrO_2 , SnO_2 , Li_2O , CeO_2 , V_2O_5 -aerogels and mixtures of these were produced (H. D. Gesser, P. C. Goswami, Chem. Rev. 1989, 89, 765 et seq.).

For some years, organic aerogels have also been known which are derived from the most widely diverse starting materials, e.g. melamine formaldehyde (R. W. Pekala, J. Mater. Sci. 1989, 24, 3221).

Inorganic aerogels can thereby be produced in different ways.

On the one hand, SiO_2 aerogels can for example be produced by acid hydrolysis and condensation of tetra-ethyl orthosilicate in ethanol. During this process, a gel is produced which can be dried by super-critical drying while its structure is maintained. Production methods based on this drying technique are known for example from EP-A-0 396 076, WO 92/03378 or WO 95/06617.

The high pressure technique involved in the super-critical drying of aerogels is however an expensive process and involves a high security risk. In addition, however, super-critical drying of aerogels is a very cost-intensive production method.

In principle, an alternative to super-critical drying is afforded by a method for the sub-critical drying of SiO_2 gels. The costs involved in sub-critical drying are substantially less by reason of the simpler technology, the lower energy costs and the lesser security risk.

The SiO_2 gels can for example be obtained by acid hydrolysis of tetra-alkoxy silanes in a suitable organic solvent by means of water. Once the solvent has been exchanged for a suitable organic solvent, the gel obtained is in a further step reacted with a silylating agent. The SiO_2 gel resulting from this can then, from an organic solvent, be dried in air. Thus, aerogels with densities of less than 0.4 g/cm³ and porosities above 60% can be achieved.

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The production method based on this drying technique is described in detail in WO 94/25149.

Furthermore, the above-described gels can, prior to drying and in the alcohol-aqueous solution, be mixed with tetra-alkoxy silanes and aged, in order to increase the gel lattice strength, as disclosed in WO 92/20623.

The tetra-alkoxy silanes used as starting materials in the above-described processes do, however, likewise represent an extremely high cost factor

A considerable cost reduction can be achieved by using water-glass as a starting material for the production of SiO_2 gels. To this end, it is possible for example to produce a silicic acid from an aqueous water-glass solution with the help of an ion exchange resin, the silicic acid then being polycondensed by the addition of a base to produce an SiO_2 gel. After exchange of the aqueous medium for a suitable organic solvent, it is then possible in a further step to react the resulting gel with a silylating agent containing chlorine. The SiO_2 gel which is surface modified for example with methyl silyl groups can then and likewise from an organic solvent, be dried in air. The production method based on this technique is known from DE-A-43 42 548.

Alternative methods with regard to the production of an SiO_2 aerogel on a basis of water-glass with subsequent sub-critical drying are described in German Patent Application 195 41 715.1 and 195 41 992.8.

Disclosed in German Patent Application No. 196 48 798.6 is a method of producing aerogels in which hydrogels are surface modified without prior solvent exchange, i.e. with essentially water in the pores, after which they are dried.

From DE-PS 896 189, it is known that globular silicic acid hydrogels can be produced by producing from a raw material, e.g. water glass, which contains silicic acid, and by reaction with an acid, for example sulphuric acid, a gel-forming silicic acid hydrosol which

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is then, in the form of individual drops, passed through a gaseous or liquid medium, e.g. a mineral oil, which is not miscible with water and the hydrosol. The hydrosol drops thereby acquire a more or less globular form and remain in the oil layer sufficiently long for the conversion from the sol to the solid hydrogel to take place. The hydrogel base produced by the indicated method do however contain mineral oil contaminations which cannot be completely eliminated even by very expensive washing processes.

In the case of this method, if the mixture is injected into a gaseous medium, then the procedure adopted is one whereby firstly hydrosol droplets are produced by a mixing jet from water-glass, sulphuric acid and aluminium sulphate, these droplets then being injected into an air-filled vessel. Under the conditions applied, conversion of the hydrosol into a hydrogel occurs within about 1 second so that the tiny hydrogel droplets can be trapped in a layer of water in the bottom of the vessel and further processed.

DE-C-21 03 243 describes a method of and an apparatus for producing substantially globular hydrogels which contain silicic acid, the silicic acid hydrosol being formed in a special mixing jet from a raw material containing silicic acid and an acid solution. The hydrosol thus formed is, for the purpose of drop formation, sprayed into a gaseous medium which does not noticeably dissolve in the hydrosol, for example air.

By reason of the necessary fall time as a reaction time for gel formation, however, dependent upon the particle size, so the overall height for the appliance into which the hydrosol is injected is unfortunately considerable. As a result, this prior art method is very cost intensive since correspondingly substantial material is required, the space requirement is considerable and it takes longer to produce the apparatus. Furthermore, in the case of this known method, hydrogel particles of a non-homogeneous size distribution arise which possibly then have to be sorted according to their size so that all in all this method becomes more time and cost intensive.

Common to all the aforesaid methods is that in order to initiate gel formation, two or more starting components, e.g. water-glass solution and mineral acid, have to be brought

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together. In this respect, it is apparently favourable for the properties of the gel particles, particularly for their subsequent stability, if the form and size of the particles can be adjusted already prior to the gel formation process. It is in particular advantageous for the subsequent stages of the process following gel formation and shaping, such as for example for washing, for possibly subsequent reactions and for the later drying process, if the particles are present in an easily-handled form, in other words for example as balls. Globular particles are in terms of stability, superior to all other shapes. By virtue of the regular geometry and the lack of edges and corners, it is possible substantially to avoid undesirable abrasion during the following stages of the process. Substantially globular lyogels have the advantage that the particle size distribution of the end product produced from the lyogel can be adjusted particularly easily by the shaping process.

Therefore, the present invention is based on the problem of providing a method of producing substantially globular lyogels in which the disadvantages of the prior art methods are avoided.

This problem is resolved in that the gel forming components are mixed together to form a lysol and then the lysol is introduced into a moving medium for forming the lyogel, the said medium being not noticeable in the lysol.

In the present Application, the term lysol or lyogel must be understood to mean a sol or a gel in which the sol or gel interstices are filled with fluid. If the fluid consists essentially of water, then one speaks of a hydrosol or hydrogel, as the case may be.

Incorporation into a moving atmosphere greatly increases the dwell time of the lysol particles in the medium so that the overall height of the appliance can be markedly reduced. Therefore, the appliances or apparatus require considerably less material and space so that the costs of the method according to the invention are considerably reduced.

Ideally, the medium is an air atmosphere, whereby further substances can be added to the lysol before it is introduced into the air atmosphere. The air can thereby also contain

other gaseous media. Any apparatuses known to a man skilled in the art for this purpose can be used for mixing the gel forming component and for incorporating the lyosol.

Expediently, the lyosol is dripped or sprayed into the air, preferably in the direction of gravity.

According to a preferred embodiment, the lyosol is added to an air stream which flows substantially against the direction of gravity. The air flow can also contain otherwise directed velocity components. Thus, the dwell time of the particles in the air can be increased under control, which results in a further saving on the overall height of the apparatus into which the lyosol is introduced. The air flow which is opposite to the direction of gravity can furthermore be used for any grading or classifying of the drops or particles during gel formation. Particles with a diameter below the limit grain diameter which corresponds to the flow velocity are delivered upwardly whereas larger particles are delivered downwardly. Consequently, no additional step is needed for sorting the gel particles according to their size so that the costs of the method according to the invention are still further reduced.

A further development of this embodiment provides for the lyosol to be introduced into an air flow, the velocity of which diminishes in the direction of flow.

A further preferred embodiment of the method resides in that the lyosol droplets, after being converted to lyogel, are trapped in a layer of water.

A further effect of the reduced falling rate of the balls is due to the air flowing against the direction of fall; the effect is one of softening the introduction of the lyogel balls in a layer of water for example.

Suitable starting substances for the method according to the invention are basically any which are usable for the prior art ways of synthesising lyogels, for example as a pre-stage for an aerogel (see for example J. Brinker, G. W. Schere, Sol-Gel Science; The Physics

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and Chemistry of Sol/Gel Processing, Academic Press Ltd., London 1990; DE-A-43 42 548; US-A-5 081 163; US-A-4 873 218).

Preferred thereby are the pre-stages of SiO_2 hydrosols, e.g. silicic acid and mineral acid. Sodium water-glass solutions and hydrochloric acid are particularly preferred.

A further problem on which the present invention is based resides in providing a method of producing substantially globular aerogels. This problem is resolved by a method in which a substantially globular lyogel, as can be produced in accordance with the present invention, is converted to an aerogel.

The method of converting the lyogel to an aerogel is in no way limited. Any alternative methods known to a man skilled in the art can be applied.

In a preferred embodiment, the substantially globular lyogel is reacted with a silylating agent. Any silylating agents such as for example trimethyl chlorosilane, which are known to a man skilled in the art, may be used. Prior to silylation, the lyogels can be washed and/or the solvent of the lyogel may be exchanged for another organic solvent. Washing of the lyogel or hydrogel and the solvent exchange can also be carried out by any methods described in the state of the art.

Drying can likewise be carried out by any methods known to a man skilled in the art. In this respect, super-critical as well as sub-critical drying processes known for aerogels are preferred, sub-critically drying being particularly preferred.

The method according to the invention is described in greater detail hereinafter with reference to an example of embodiment.

Example 1

A sodium water-glass solution is produced by dilution of 53.5 kg commercially available sodium water-glass solution with 25.5% SiO_2 and 7.6% Na_2O with 31.7 kg of deionised

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water. A dilute hydrochloric acid is produced by dilution of 19.3 kg commercially available 25% hydrochloric acid with 65.8 kg deionised water. In each case 30 kg/hr of the dilute hydrochloric acid and of the dilute water-glass solution are fed to a mixing and spraying apparatus in precisely measured amounts. The outlet of the mixing nozzle is situated at the top end of a pipe through which heated air flows vertically upwards. The bottom third of the pipe is filled with water. Above the surface of the water, the pipe has air inlet apertures. The flow of air is adjusted to an empty pipe velocity of 4 m/sec. The temperature inside the pipe is 100°C. The hydrogel spheres are captured in the water layer, settle through the layer of water and are delivered from the spray tower in a stream of water.

The small hydrogel balls are continuously washed with 0.1 mol hydrochloric acid and then with deionised water. Subsequently, the lyogel balls are washed with acetone in several stages until the water content in the gel is less than 1%. The acetone-moist gel is exposed to a mixture of acetone and 5% trimethyl chlorosilane for 10 hours. Then, again in several stages, the gel is washed with acetone. The acetone-moist gel balls are dried in a fluidised bed with nitrogen at 180°C for 5 mins. The aerogel balls obtained have a density of 130 kg/cu.m and their heat conductivity is 0.01 W/mK.

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The following statements present various aspects and features of the present invention:

1. A method of producing substantially globular lyogels in which the gel forming components are blended to form a lyosol after which, in order to form the lyogel, the lyosol is introduced into the moving medium which does not dissolve noticeably in the lyosol.
2. A method according to statement 1, characterised in that the medium is air.
3. A method according to statement 2, characterised in that the air contains at least one further gaseous medium.
4. A method according to statement 2 or 3, characterised in that the lyosol is fed dropwise into the moving air.
5. A method according to statement 2 or 3, characterised in that the lyosol is sprayed into the moving air.
6. A method according to at least one of statements 2 to 5, characterised in that the lyosol is introduced into a stream of air directed substantially in opposition to the force of gravity.
7. A method according to statement 6, characterised in that the lyosol particles are screened according to size by the flow of air which is directed in opposition to the force of gravity.
8. A method according to statement 6 or 7, characterised in that the velocity of the air flow diminishes in the direction of flow.
9. A method according to at least one of the preceding statements, characterised in that the lyosol particles are trapped in a water layer.

10. A method according to at least one of the preceding statements, characterised in that the lyosol consists of silicic acid and mineral acid.
11. A method according to at least one of statements 1 to 9, characterised in that the lyosol is formed from a sodium water-glass solution and hydrochloric acid.
12. Use of substantially globular lyogels such as are obtainable in accordance with at least one of the preceding statements, in order to produce aerogels.
13. A method of producing substantially globular aerogels in which a substantially globular lyogel, obtained in accordance with at least one of statements 1 to 11, is converted to an aerogel.

The following additional statements present various aspects and features of the present invention:

1. A method of producing substantially globular lyogels in which the gel forming components are mixed to produce a lyosol, after which the lyosol, in order to form a lyogel, is introduced into a moving medium which flows substantially against the direction of the force of gravity and which does not perceptibly dissolve in the lyosol.
2. A method according to additional statement 1, characterised in that the medium is air.
3. A method according to additional statement 2, characterised in that the air contains at least one further gaseous medium.
4. A method according to additional statement 2 or 3, characterised in that the lyosol is introduced dropwise into the moving air.
5. A method according to additional statement 2 or 3, characterised in that the lyosol is sprayed into the moving air.
6. A method according to at least one of additional statements 2 to 5, characterised in that the lyosol particles are screened according to size by the air stream which is directed in opposition to the force of gravity.
7. A method according to at least one of additional statements 2 to 5, characterised in that the velocity of the air stream diminishes in the direction of flow.
8. A method according to at least one of the preceding additional statements, characterised in that the lyosol particles are trapped in a layer of water.

9. A method according to at least one of the preceding additional statements, characterised in that the lyosol is formed from silicic acid and mineral acid.
10. A method according to at least one of additional statements 1 to 8, characterised in that the lyosol is formed from a sodium water-glass solution and hydrochloric acid.
11. Use of substantially globular lyogels, produced according to at least one of the preceding additional statements, for the production of aerogels.
12. A method of producing substantially globular aerogels in which a substantially globular lyogel, produced according to at least one of additional statements 1 to 10, is converted to an aerogel.

What is claimed is:

- 1 1. A method of producing substantially globular lyogels in which the gel forming
2 components are mixed to produce a lyosol, after which the lyosol, in order to form a lyogel, is
3 introduced into a moving medium which flows substantially against the direction of the force
4 of gravity and which does not perceptibly dissolve in the lyosol.
- 1 2. A method according to claim 1, characterised in that the medium is air.
- 1 3. A method according to claim 2, characterised in that the air contains at least one
2 further gaseous medium.
- 1 4. A method according to claim 2, characterised in that the lyosol is introduced dropwise
2 into the moving air.
- 1 5. A method according to claim 2, characterised in that the lyosol is sprayed into the
2 moving air.
- 1 6. A method according to at least one of claim 2, characterised in that the lyosol
2 particles are screened according to size by the air stream which is directed in opposition to
3 the force of gravity.
- 1 7. A method according to at least one of claim 2, characterised in that the velocity of the
2 air stream diminishes in the direction of flow.
- 1 8. A method according to claim 1, characterised in that the lyosol particles are trapped in
2 a layer of water.

DECLARATION AND POWER OF ATTORNEY - ORIGINAL APPLICATION

Attorney's Docket No.
AE97/151US

My residence, post office address and citizenship are as stated below next to my name;

I verily believe I am the original, first and sole inventor (if only one name is listed below at 201) or an original, first and joint inventor (if plural inventors are named below at 201-203) of the subject matter which is claimed and for which a patent is sought on the invention entitled **Method for Producing Substantially Globular Lyogels and Aerogels** the specification of which is attached hereto.

I have reviewed and understand the contents of the above-identified specification, including the claims. I acknowledge my duty to disclose information of which I am aware which is material to the examination of this application in accordance with Section 1.56(a), Title 37 of the Code of Federal Regulations; and as to applications for patents or inventors certificate on the invention filed in any country foreign to the United States of America, prior to this application by me or my legal representatives or assigns,

☐ no such applications have been filed, or
☒ such applications have been filed as follows:

Country	Application No.	Date of Filing (day,month,yr)	Date of Issue	Priority Claimed Under 35 USC 119			
Germany	DE 19722738.4	May 30, 1997		YES	X	NO	
PCT	EP/98/03161	May 28, 1998		YES	X	NO	

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the United States Patent and Trademark Office connected therewith and to act on my behalf before the competent International Authorities in connection with any and all international applications filed by me. (List name and registration number)

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as my Attorneys

SC N NED 7

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 201

DATE

SIGNATURE OF INVENTOR 202

DATE

SIGNATURE OF INVENTOR 203

DATE